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**(54) Resin-coated proppant for subterranean fractures**

(57) Resin-coated proppant particles are suspended in a fracturing fluid and deposited in a subterranean fracture and consolidated into high strength permeable masses. As the fractures are formed, a liquid hardenable resin component is continuously mixed with a liquid hardening agent component on-the-fly to form a hardenable resin composition. The hardenable resin composition

is continuously coated onto dry proppant particles on-the-fly to form resin composition coated proppant particles, and the resin composition coated proppant particles are continuously suspended in the fracturing fluid on-the-fly.

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[0006] The present invention provides improved methods of coating dry proppant particles with a resin composition and suspending the coated proppant particles in a fracturing fluid. The resin composition hardens and consolidates the resin coated proppant particles into high strength permeable packs in fractures formed in subterranean zones which do not allow proppant flow-back. The methods are basically comprised of the following steps. A liquid hardenable resin component is provided comprised of a hardenable resin, a silane coupling agent, a solvent for the resin, a hydrolyzable ester for breaking gelled fracturing fluid films on the proppant particles, and a surfactant facilitating the coating of the resin on the proppant particles and for causing the hardenable resin to flow to the contact points between adjacent resin coated proppant particles so that the particles are consolidated into a high strength permeable mass. A liquid hardening agent component is provided comprised of a hardening agent suspended or dissolved in a liquid carrier fluid. In addition, a source of dry proppant particles and a gelled liquid fracturing fluid are provided. The gelled liquid fracturing fluid is pumped into a subterranean zone to form one or more fractures therein and to place resin composition coated proppant particles in the fractures. As the fractures are formed by the fracturing fluid, the liquid hardenable resin component is mixed with the liquid hardening agent component to form a resin composition. The resin composition is continuously coated on dry proppant particles conveyed from the source of the dry proppant particles. The resulting resin composition coated proppant particles are continuously mixed with the fracturing fluid whereby the resin composition coated proppant particles are suspended in the fracturing fluid and are deposited in the one or more fractures formed wherein they are caused to harden and consolidate into a high strength permeable pack which prevents proppant flow-back. When the resin composition coated proppant particles have been placed in the one or more fractures, the pumping of the fracturing fluid, the mixing of the liquid hardenable resin component with the liquid hardening agent component, the coating of the dry proppant particles with the resin composition and the mixing of the resin composition coated proppant particles with the fracturing fluid are terminated. The hardenable resin composition on the coated proppant particles is allowed to harden and to consolidate the proppant into one or more high strength permeable packs which prevent proppant flow-back.

[0007] Another improved method of the present invention for coating dry proppant particles with a resin composition and suspending the coated proppant particles in a fracturing fluid whereby the resin coated proppant particles are placed in one or more fractures formed in a subterranean zone by the fracturing fluid is comprised of the following steps. A liquid hardenable resin component as described above and a liquid hardening agent component as described above are provided along with a source of dry proppant particles and a gelled liquid fracturing fluid. The gelled liquid fracturing fluid is pumped into the subterranean zone to form one or more fractures therein and to place the resin composition coated proppant particles in the fractures. The liquid hardenable resin component is continuously mixed with the liquid hardening agent component to form a resin composition. The volume ratio of the liquid hardening agent component to the liquid hardenable resin component is varied from an initial volume ratio to a lower volume ratio and then back to the initial volume ratio in order to consolidate all of the proppant particles in the fractures while conserving the amount of the liquid hardening agent component used. The resin composition is continuously coated onto dry proppant particles conveyed from the source thereof to form resin composition coated proppant particles. The resin composition coated proppant particles are continuously mixed with the fracturing fluid so that the resin composition coated proppant particles are continuously deposited in the fractures. Thereafter, the pumping of the fracturing fluid into the subterranean zone, the mixing of the liquid hardenable resin component with the liquid hardening agent component, the coating of the resin composition onto the dry proppant particles to form resin composition coated proppant particles and the mixing of the resin composition coated proppant particles with the fracturing fluid are terminated. The hardenable resin composition on the coated proppant particles is allowed to harden and to consolidate the proppant into one or more high strength permeable packs which prevent proppant flow-back.

[0008] As mentioned above, the prior art methods of consolidating proppant in subterranean fractures have generally included the placement of a large quantity of uncoated proppant in the fractures followed by a tail-end portion of proppant coated with a hardenable resin composition. When the viscous fracturing fluid which is the carrier for the proppant is broken and reverts to a thin fluid, the resin coated proppant is deposited in the fractures. The tail-end portion of the resin coated proppant is consolidated into a hard permeable mass by the resin composition, but the uncoated proppant previously deposited in the bottom of the fractures as the large quantity of uncoated proppant was transported into the fractures is not consolidated. As a result, when the well is placed on production, flow-back of the uncoated proppant takes place.

[0009] Another problem which is encountered in consolidating the proppant to prevent flow-back involves the strength of the consolidated resin coated proppant packs in the fractures. Heretofore, it has been a common practice to utilize proppant that is precoated with a resin composition and stored. When the precoated proppant is required for use at a job site, it is transported to the job site. While such precoated resin coated proppant is consolidated after being placed by a hardening agent, the resulting consolidated proppant pack often does not have enough strength to prevent deterioration and proppant flow-back.

[0010] In accordance with the present invention, all of the proppant transported into the fractures is coated with a resin composition on-the-fly as the fracturing fluid is pumped into the well bore and into the fractures. As a result, the

the cost of the fracturing procedure, the volume ratio of the liquid hardening agent component can be varied. That is, in a preferred technique, the volume ratio of the liquid hardening agent component to the liquid hardenable resin component is varied from an initial volume ratio which produces a high strength permeable pack to a lower volume ratio which produces a lower strength resilient permeable pack and then back to the initial volume ratio which produces a high strength permeable pack adjacent to the well bore. As mentioned, because all of the proppant particles are coated with the liquid hardening agent component, there is no unconsolidated proppant particles in the bottoms of the fracture and consequently, the consolidated permeable pack does not allow flow-back to occur. The initial volume ratio of the liquid hardening agent component to the liquid hardenable resin component is generally in the range of from about 1:100 to about 1:2 and the lower volume ratio is in the range of from 0 to about 1:5. Preferably, the initial volume ratio of the liquid hardening agent component to the liquid hardenable resin component is about 1:2 and the lower volume ratio is about 1:10.

**[0014]** Thus, another method of the present invention includes the following steps. A liquid hardenable resin component is provided comprised of a hardenable resin, a silane coupling agent, a solvent for the resin, one or more hydrolyzable esters for breaking gelled fracturing fluid films on the proppant particles, and a surfactant for facilitating the coating of the resin on the proppant particles and for causing the hardenable resin to flow to the contact points between adjacent resin coated proppant particles. A liquid hardening agent component is provided comprised of a hardening agent suspended or dissolved in a liquid carrier fluid. In addition, a source of dry proppant particles and a gelled liquid fracturing fluid are provided. The gelled liquid fracturing fluid is pumped into the subterranean zone to form one or more fractures therein and to place the resin composition coated proppant particles in the fractures. As the fractures are formed the liquid hardenable resin component is continuously mixed with the liquid hardening agent component on-the-fly to form the resin composition. The volume ratio of the liquid hardening agent component to the liquid hardenable resin component is varied, and the resin composition produced is continuously coated onto dry proppant particles conveyed from the source thereof on-the-fly to form resin composition coated proppant particles. The resin composition coated proppant particles are continuously mixed with the fracturing fluid being pumped on-the-fly whereby the resin composition coated proppant particles are suspended therein. After the resin composition coated proppant particles have been placed in one or more fractures formed in the subterranean zone, the pumping of the gelled liquid fracturing fluid and other related steps are terminated. Thereafter, the gelled liquid fracturing fluid breaks into a thin fluid, the resin composition coated proppant particles are deposited in the fractures and the resin composition hardens and consolidates the proppant particles in one or more fractures into high strength permeable packs which do not allow proppant flow-back.

**[0015]** Examples of hardenable resins which can be utilized in the liquid hardenable resin component include, but are not limited to, organic resins such as bisphenol A-epichlorohydrin resin, polyepoxide resin, novolak resin, polyester resin, phenol-aldehyde resin, urea-aldehyde resin, furan resin, urethane resin and mixtures thereof. Of these, bisphenol A-epichlorohydrin resin is preferred. The organic resin utilized is included in the liquid hardenable resin component in an amount in the range of from about 50% to about 90% by weight of the liquid hardenable resin component, preferably an amount of about 85%.

**[0016]** Examples of silane coupling agents which can be used include, but are not limited to, N-2-(aminoethyl)-3-aminopropyltrimethoxysilane and 3-glycidoxypropyl-trimethoxysilane. Of these, 3-glycidoxypropyltrimethoxysilane is preferred. The silane coupling agent is included in the liquid hardenable resin component in an amount in the range of from 0% to about 5% by weight of the liquid hardenable resin component, preferably in an amount of about 2%.

**[0017]** Examples of solvents for the organic resin and silane coupling agent in the liquid hardenable resin component include, but are not limited to, methanol, butyl alcohol and N-methyl-2-pyrrolidone. Of these, methanol is preferred. The amount of the solvent utilized in the liquid hardenable resin component is in the range of from about 1% to about 10% by weight of the liquid hardenable resin component, preferably in an amount of about 5%.

**[0018]** Examples of hydrolyzable esters utilized in the liquid hardenable component for facilitating the coating of the resin composition on the proppant particles and for breaking gelled fracturing fluid films thereon, include, but are not limited to, a mixture of dimethylglutarate, dimethyladipate and dimethylsuccinate, sorbitol, catechol, dimethylthiolate and ter butylhydroperoxide. Of these, a mixture dimethylglutarate, dimethyladipate and dimethylsuccinate is preferred. The hydrolyzable ester is present in the liquid hardenable component in an amount in the range of from about 0.2% to about 2 percent by weight of the liquid hardenable resin component, preferably in an amount of about 1.2%.

**[0019]** The surfactants which can be utilized in the liquid hardenable resin component for facilitating the coating of the resin on the proppant particles, and for causing the hardenable resin to flow to the contact points between adjacent resin coated proppant particles include, but are not limited to, an ethoxylated nonyl phenol phosphate ester and mixtures of one or more cationic surface active agents and one or more non-ionic surface active agents. Such mixtures are described in U.S. Patent No. 6,311,733 issued to Todd et al. on November 6, 2001 which is incorporated herein by reference. An ethoxylated nonyl phenol phosphate ester is preferred. The surfactant or surfactants utilized are included in the liquid hardenable resin component in an amount in the range of from about 1% to about 15% by weight of the liquid hardenable resin component, preferably in an amount of about 8%.

formation particulate solids which migrate with produced fluids are prevented from being produced from the subterranean zone. Various kinds of proppant can be utilized including graded sand, bauxite, ceramic materials, glass materials and the like. Generally, the proppant particles have a size in the range of from about 2 to about 400 mesh, U.S. Sieve Series. The preferred proppant is graded sand having a particle size in the range of from about 10 to about 70 mesh, U.S. Sieve Series. Preferred sand particle size distribution ranges are one or more of 10-20 mesh, 20-40 mesh, 40-60 mesh or 50-70 mesh, depending on the particular size and distribution of formation solids to be screened out by the consolidated proppant particles.

[0030] A preferred improved method of this invention for coating dry proppant particles with a hardenable resin composition and suspending the coated proppant particles in a fracturing fluid, the resin composition hardening and consolidating the resin coated proppant particles into a high strength permeable mass which prevents proppant flow-back after being placed in one or more fractures in a subterranean zone comprising the steps of: (a) providing a liquid hardenable resin component comprised of a hardenable resin, a silane coupling agent, a solvent for the resin, one or more hydrolyzable esters for breaking gelled fracturing fluid films on the proppant particles, and a surfactant for facilitating the coating of the resin on the proppant particles and for causing the hardenable resin to flow to the contact points between adjacent resin coated proppant particles; (b) providing a liquid hardening agent component comprised of a hardening agent suspended or dissolved in a liquid carrier fluid; (c) providing a source of dry proppant particles; (d) providing a gelled liquid fracturing fluid; (e) pumping the gelled liquid fracturing fluid into the subterranean zone to form the one or more fractures therein and to place the hardenable resin composition coated proppant particles in the fractures; (f) as the fractures are formed in step (e), continuously mixing the liquid hardenable resin component with the liquid hardening agent component on-the-fly to form the hardenable resin composition; (g) continuously coating the hardenable resin composition produced in step (f) onto dry proppant particles conveyed from the source thereof on-the-fly to form resin composition coated proppant particles; (h) continuously mixing the resin composition coated proppant particles produced in step (g) with the fracturing fluid pumped in accordance with step (e) on-the-fly whereby the resin composition coated proppant particles are suspended therein; (i) terminating steps (e), (f), (g) and (h) when the resin composition coated proppant particles have been placed in the one or more fractures; and (j) allowing the hardenable resin composition on the resin composition coated proppant particles to harden and consolidate the proppant particles into one or more high strength permeable packs which prevent proppant flow-back.

[0031] Another preferred improved method of coating dry proppant particles with a hardenable resin composition and suspending the coated proppant particles in a fracturing fluid, the resin composition hardening and consolidating the resin coated proppant particles into a high strength permeable mass which prevents proppant flow-back after being placed in one or more fractures formed in a subterranean zone comprising the steps of: (a) providing a liquid hardenable resin component comprised of a hardenable resin, a silane coupling agent, a solvent for the resin, one or more hydrolyzable esters for breaking gelled fracturing fluid films on the proppant particles, and a surfactant for facilitating the coating of the resin on the proppant particles and for causing the hardenable resin to flow to the contact points between adjacent resin coated proppant particles; (b) providing a liquid hardening agent component comprised of a hardening agent suspended or dissolved in a liquid carrier fluid; (c) providing a source of dry proppant particles; (d) providing a gelled liquid fracturing fluid; (e) pumping the gelled liquid fracturing fluid into the subterranean zone to form the one or more fractures therein and to place the resin composition coated proppant particles in the fractures; (f) as the fractures are formed in step (e), continuously mixing the liquid hardenable resin component with the liquid hardening agent component on-the-fly to form the hardenable resin composition; (g) varying the volume ratio of the liquid hardening agent component to the liquid hardenable resin component during step (f); (h) continuously coating the hardenable resin composition produced in steps (f) and (g) onto dry proppant particles conveyed from the source thereof on-the-fly to form resin composition coated proppant particles; (i) continuously mixing the resin composition coated proppant particles produced in step (h) with the fracturing fluid pumped in accordance with step (e) on-the-fly whereby the resin composition coated proppant particles are suspended therein; (j) terminating steps (e), (f), (g), (h) and (i) when the resin composition coated proppant particles have been placed in the one or more fractures; and (k) allowing the hardenable resin composition on the resin composition coated proppant particles to harden and consolidate the proppant particles into one or more high strength permeable packs which prevent proppant flow-back.

[0032] In order to further illustrate the methods of the present invention, the following examples are given.

#### Example 1

[0033] To determine the effect of resin concentration on the consolidation strengths of proppant packs, bauxite proppant was dry coated with various amounts of mixed resin compositions. The concentration is expressed in terms of weight percent of proppant. After dry coating with the mixed resin, the proppant was mixed with a cross-linked 30 pound per 1,000 gallon linear carboxymethyl guar fracturing fluid, poured into a consistometer, stirred for 60 minutes at 175°F to simulate pumping, transferred to flow cells, packed, and cured in an oven at 300°F for 3 hours with and without 500-psi closure stress. A consistometer is a device designed to stir a solid containing slurry at a desired temperature.

TABLE III

Effect Of Simulated Pump Time On Consolidation			
Test No.	Description Of Test	Unconfined Compressive Strength Without Closure Pressure, psi	Unconfined Compressive Strength With Closure Pressure, psi
1	Stirred in consistometer for 30 min. at 175°F. Stopped stirring and remained at 175°F for 3 hours. Packed cross-linked slurry into flow cells and cured at 300°F for 3 hours without closure applied. Stirred in consistometer for 30 min. at 175°F.	1410	
2	Packed in flow cell without closure, remained in heat bath at 175°F for 3 hours. Applied 500 psi closure, cured at 300°F for 3 hours.		1945
3	Stirred in consistometer for 1 hour at 175°F. Packed and cured at 300°F for 3 hours.	1560	1690
4	Stirred in consistometer for 1 hour at 175°F. Packed and cured at 300°F for 3 hours.	1790	1930
5	Stirred in consistometer for 1 hour at 175°F. Packed and cured at 300°F for 3 hours.	1680	1795

**Example 4**

[0036] The procedure of Example 2 was repeated except that portions of the proppant were dry coated with resin compositions containing varying amounts of hardening agent. Each of the resin coated proppant portions were mixed with portions of a cross-linked 30 pound per gallon linear carboxymethyl guar fracturing fluid and tested as indicated in Example 2.

TABLE IV

Effect Of Varying Amounts Of Liquid Hardening Agent Component On Consolidation and Flow-Back				
Test No.	Volume % Liquid Hardening Agent Component	Cure Time, hrs	Unconfined Compressive Strength, psi	Proppant Flow-Back Amount, grams
1	0	3	6	None
2	0	20	7	None
3	5	3	9	None
4	5	20	22	None
5	10	3	140	None
6	10	20	150	None
7	25	3	425	None
8	50	3	1155	None
9	100	3	1680	None

[0037] As indicated in Table IV, various amounts of liquid hardening agent component ranging from 0 to 100 percent of the initial amount can be mixed with the liquid hardenable resin component to achieve different degrees of rubbery/

liquid hardening agent component to said liquid hardenable resin component during the time steps (a) to (d) are carried out.

- 5 10. A method according to claim 9, wherein the volume ratio of said liquid hardening agent component to said liquid hardenable resin component is varied from an initial volume ratio, preferably of from 1:100 to 1:2, to a lower volume ratio, preferably up to 1:5, and then back to said initial volume ratio.

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# ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO.

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This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on  
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